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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF FERRO-MOLYBDENUM

PART 5 DETERMINATION OF PHOSPHORUS

भारतीय मानक

फेरो-मालिबडेनम — रासायनिक विश्लेषण की पद्धति

भाग 5 फास्फोरस ज्ञात करना

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 1989

Price Group 1

Methods of Chemical Analysis of Ferrous Metals Sectional Committee, SMDC 2

FOREWORD

This Indian Standard (Part 5) was adopted by the Bureau of Indian Standards on 17 October 1988, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

IS 1559 : 1961 'Methods of chemical analysis of ferro-alloys' covers the, chemical analysis of different ferro-alloys, such as, ferrosilicon, ferrochromium, ferromanganese and speigeleisen, silico manganese, ferro-molybdenum, ferrophosphorus, ferrotitanium, ferrovanadium and ferrotungsten. While reviewing this standard, the sectional committee had decided that a separate series for each ferroalloy be prepared. Accordingly, IS 1559 in its various parts was published for the chemical analysis of ferro silicon.

The chemical analysis of ferro-molybdenum is covered into series of parts of this standard. With the publication of these parts, the analysis of relevant constituents prescribed for ferro-molybdedum in IS 1559 : 1961 will be superseded. This part covers the determination of total carbon. The other parts are:

Part 1 Determination of molybdenum
Part 2 Determination of total carbon
Part 3 Determination of silicon
Part 4 Determination of sulphur
Part 6 Determination of copper
Part 7 Determination of aluminium

In this revision, the volumetric method for determination of phosphorus by alkalimetric method has been incorporated.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accodance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF FERRO-MOLYBDENUM

PART 5 DETERMINATION OF PHOSPHORUS

1 SCOPE

This standard (Part 5) covers the alkalimetric method for determination of phosphorus in ferro-molybdenum.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
IS 264 : 1976	Specification for nitric acid
IS 265 : 1976	Specification for hydrochloric acid
IS 1472 : 1977	Method of sampling ferro-alloys for determination of chemical composition

3 SAMPLING

Samples shall be drawn and prepared in accordance with IS 1472 : 1977.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water shall be employed in the tests.

5 DETERMINATION OF PHOSPHORUS IN FERRO-MOLYBDENUM BY ALKALIMETRIC METHOD

5.1 Outline of the Method

The sample is dissolved in acids. Phosphorus is precipitated as ammonium phosphomolybdate which is dissolved in known excess of sodium hydroxide and unused sodium hydroxide is titrated against nitric acid.

5.2 Reagents

5.2.1 *Concentrated Nitric Acid,* rd=1·42 (conforming to IS 264 : 1976)

5.2.2 *Concentrated Hydrochloric Acid,* rd=1·16 (Conforming to IS 265 : 1976)

5.2.3 *Dilute Sulphuric Acid,* 1 : 1 (*v*/*v*)

5.2.4 *Dilute Hydrochloric Acid* 1 : 1, 1 : 5 (*v*/*v*)

5.2.5 *Ammonium Hydroxide,* rd=0·90

5.2.6 *Dilute Nitric Acid* 1 : 3, 1 : 99 (*v*/*v*)

5.2.7 *Ammonium Molybdate Solution*

Add solution *A* (*see* **5.2.7.1**) slowly with constant stirring to solution *B* (*see* **5.2.7.2**) kept cool in a cold water bath. Add 10 ml of ammonium phosphate (1 g/litre) and keep the solution at least for 24 hours. Filter the solution through Whatman filter paper No. 541 before use.

5.2.7.1 *Solution A*

Dissolve 100 g of molybdic acid (MoO_3) (85 percent) or 118 g of ammonium molybdate in a mixture of 145 ml of ammonium hydroxide and 270 ml of water. Cool the solution.

5.2.7.2 *Solution B*

Add 490 ml of concentrated nitric acid to 1 150 ml of water and cool.

5.2.8 *Potassium Nitrate,* 2 percent

5.2.9 *Standard Sodium Hydroxide Solution,* (0·1 N)

Dissolve 4·5 g of sodium hydroxide in one litre of freshly boiled and cooled water, and barium hydroxide. Standardize against standard nitric acid (**5.2.10**).

5.2.10 *Standard Nitric Acid* (0·1 N)

Dilute 7 ml of concentrated nitric acid to 1 litre with freshly boiled water. Standardize against sodium carbonate.

5.3 Procedure

5.3.1 Dissolve 2·00 g of sample in 20 ml each of concentrated nitric acid and hydrochloric acid in a 250 ml beaker. Add 20 ml dilute sulphuric acid and evaporate to dense white fumes. Cool, add 75 ml of dilute hydrochloric acid (1 : 5) and boil. Cool and filter through filter paper pulp pad. Wash pulp with hot dilute hydrochloric acid (1 : 5).

5.3.2 Dilute the filtrate to 250 ml; add ammonium hydroxide in slight excess, boil for 1 to 2 minutes and allow the precipitate to settle. Filter through a loose texture paper and wash the precipitate with hot water. Discard the filtrate.

5.3.3 Dissolve the precipitate in 50 ml of hot dilute nitric acid (1 : 3) and boil to expel the oxides of nitrogen. Cool the contents to about 90°C. Add 45 ml of ammonium molybdate solution and shake vigorously for 5 min. Allow the precipitate to settle for 1 hour.

5.3.4 Filter the precipitate on the filter paper pulp pad. Wash with dilute nitric acid (1 : 99) twice, and then with potassium nitrate solution until 10 ml portion of the filtrate gives pink colour with phenolphthalein and a drop of sodium hydroxide solution.

5.3.5 Transfer the pulp pad with precipitate to a conical flask. Add 25 ml of water and excess of standard sodium hydroxide solution and shake until the precipitate is dissolved. Dilute to 100 ml with water and titrate with standard nitric acid to disappearance of pink colour.

5.3.6 *Blank* — Carry out a blank determination on the same quantity of standard sodium hydroxide solution added in **5.3.5** and titrate with standard nitric acid.

5.4 *Calculation*

$$\text{Phosphorus, percent} = \frac{(A-B) \times C \times 0{\cdot}135}{D}$$

where

A = volume in ml of standard nitric acid required for the blank,

B = volume in ml of standard nitric acid solution requirement to titrate the excess sodium hydroxide solution,

C = normality of standard nitric acid solution, and

D = mass in g of sample taken.

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